ANL/NE-16/5

High Throughput Method of Extracting and Counting Strontium-90 in Urine

Nuclear Engineering Division

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High Throughput Method of Extracting and Counting Strontium-90 in Urine

by
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Contents

Abstract

A method has been developed for the rapid extraction of Sr-90 from the urine of individuals exposed to radiation in a terrorist attack. The method employs two chromatographic ion-exchange materials: Diphonix resin and Sr resin, both of which are commercially available. The Diphonix resin reduces the alkali ion concentrations below 10 mM, and the Sr resin concentrates and decontaminates strontium-90. Experimental and calculational data are given for a variety of test conditions. On the basis of these results, a flowsheet has been developed for the rapid concentration and extraction of Sr-90 from human urine samples for subsequent beta-counting.

1. Introduction

An analytical method for the rapid extraction of Sr-90 from urine is being developed for the evaluation of individuals contaminated with radionuclides from a radiological terrorist event. This method will allow rapid evaluation and screening of individuals for possible exposure to Sr-90 by a critical medical management team and will allow contaminated individuals to be administered chemical countermeasures in the critical time frame after initial exposure.

The suggested approach is based on using two chromatographic ion-exchange materials: Diphonix resin and Sr resin (both obtained from Eichrom). Initial experimental results are discussed in an earlier report by Hawkins et al.[1] These two resins exhibit opposite trends in the dependence of the corresponding distribution ratios for Sr^{2+} (D_{Sr}, which is the ratio of concentrations of the ions in the solid and liquid phases) as a function of the acidity of the carrier phase (Figures 1 and 2a). This property allows one to use columns containing these two resins in tandem, as the high acidity that facilitates stripping of ^{90}Sr from the Diphonix resin column is optimum for loading strontium on the Sr resin column. Aqueous methane sulfonic acid (MSA) solutions were chosen as the carrier phase for the Diphonix resin, as the presence of this acid (< 4 M) has no effect on ^{90}Sr uptake by Sr resin (only the concentration of the nitric acid affects Sr separations), while for Diphonix resin, only the overall proticity determines D_{Sr} (the same concentrations of the nitric acid and MSA have the same effect on ion retention). Since nitric acid tends to nitrate aromatic constituents in urine, resulting in strong coloration (even when these components are present at low concentration), the contact with strong nitric acid needs be

[1] C. Hawkins, M. Dietz, M. Kaminski, C. Mertz, and I. Shkrob, "Towards a Method of Rapid Extraction of Strontium-90 from Urine: Urine Pretreatment and Alkali Metal Removal," Argonne National Laboratory Report ANL/NE-16/2, March 2016.

delayed until the ^{90}Sr is preconcentrated and most of the organic components are removed from the urine matrix.

Strontium resin is a polyacrylic material (Figure 1) impregnated with derivatized 18-crown-6 ionophore in 1-octanol. As this crown ether has affinity for potassium and sodium, addition of > 10 mM K^+ or > 100 mM Na^+ considerably reduces the retention of Sr^{2+}. Furthermore, the resin has a finite ion capacity that should not be exceeded. Since the urine samples have 100-150 mM sodium and 50-100 mM potassium, the retention of Sr^{2+} in such saline solutions is greatly reduced. Our approach is using the Diphonix resin to remove these K^+ and Na^+ interferences by reducing the alkali ion concentrations below 10 mM and then using the minimal-volume Sr resin column to concentrate and decontaminate preconcentrated strontium-90.

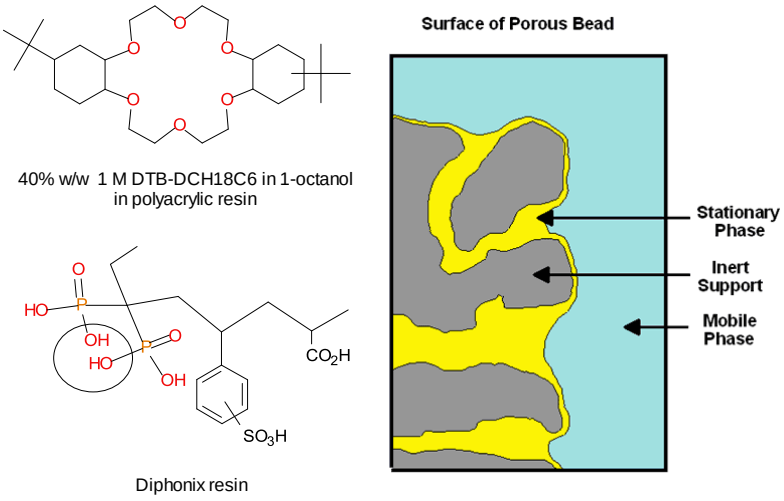

Figure 1. (Left) Structural formulas of strontium and Diphonix ion-exchange resins. (Right) Schematic depiction of the impregnated porous Sr resin; the beads are 50-100 μm; the pores are 10-20 nm.

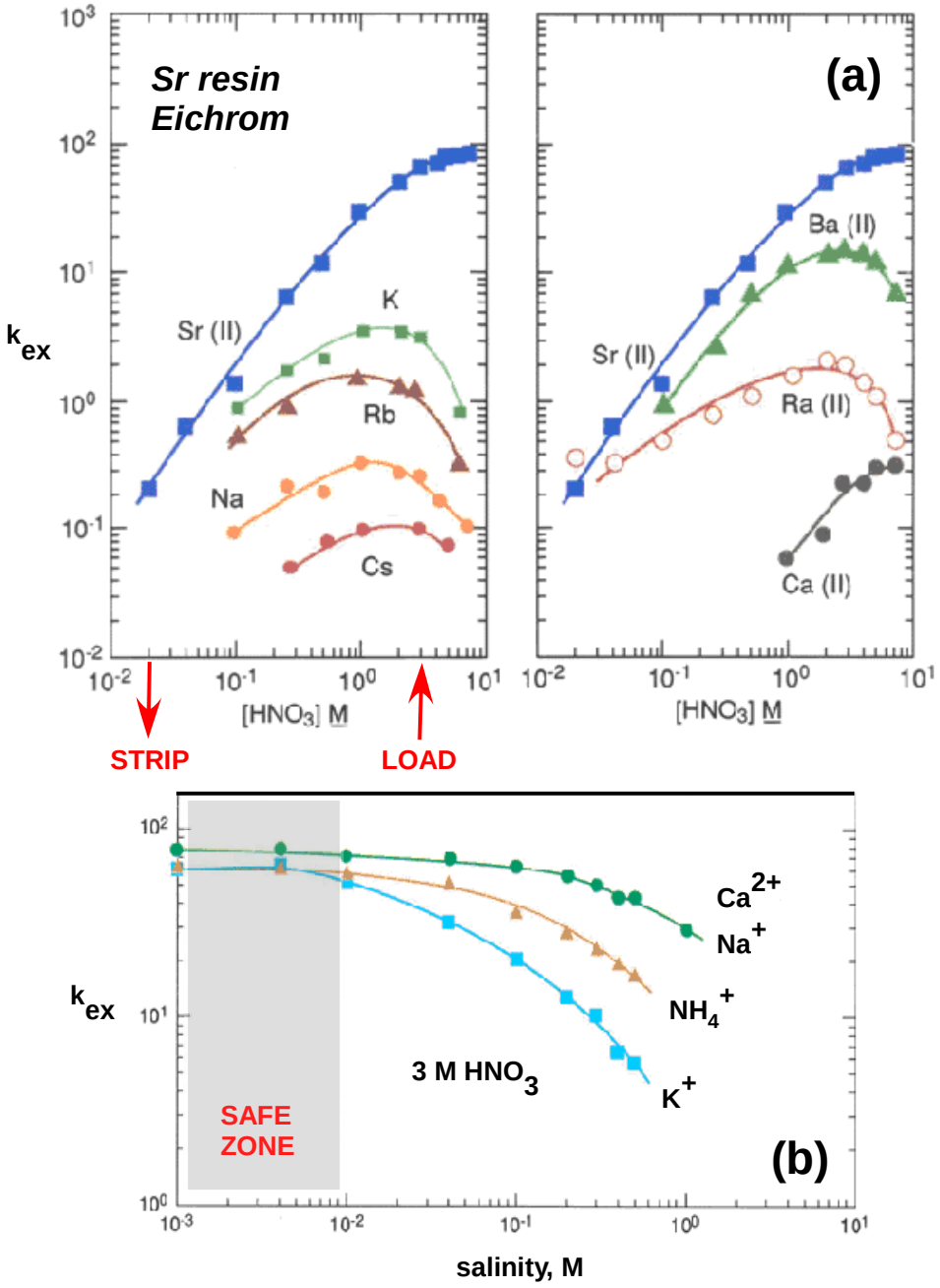

Figure 2. Coefficients k_{ex} obtained with Sr resin (Eichrom) for (a) Sr^{2+} and other ions as a function of nitric acid concentration and (b) Na^+, K^+, Ca^{2+}, and NH_4^+ as a function of salinity in 3 M HNO_3. These two plots were provided by the manufacturer of this resin. The "safe zone" indicates the salinity region conducive for maximal concentration of ^{90}Sr on the column.

2. Process Retention Characterization

The retention of a given ion on a chromatographic column (with an infinite number of theoretical plates) operating under quasi-equilibrium conditions is characterized by the constant k_{ex}, corresponding to the elution volume (in column volumes) required for the peak concentration (for a small loaded sample) to reach the opposite end of the column during the elution. The same value of k_{ex} gives the approximate load volume under which such a column would not break through for a given ion. While real ion-exchange columns have a finite number of theoretical plates and the conditions may not correspond to quasi-equilibrium, k_{ex} is a convenient parameter characterizing column operation. The coefficient k_{ex} is related to the weight distribution coefficient D_w through

$$k_{ex} = D_w \rho V_s/V_m, \quad (1)$$

where ρ is the density of the resin, V_s is the volume of the stationary phase, and V_m is the volume of the mobile phase, so that $V_s/(V_s+V_m)$ is the bed ratio. Using ρ=1.16 g/mL and a bed ratio of 0.3 (manufacturers recommended values, see http://www.eichrom.com/radiochem) for the Diphonix resin yields $k_{ex} \approx D_w/2.01$. We calculated the retention coefficient k_{ex} for Sr^{2+}, K^+, and Na^+ with the Diphonix resin by substituting the data given in Figure 2 into Eq. 1. The calculated k_{ex} is plotted as a function of the acidity of the mobile phase (the MSA concentration) in Figure 3. Referencing the concentration to the standard conditions (chosen as 0.1 M MSA) for the aqueous solution yields

$$k_{ex}/k_{ex}^{o} \approx Q^{\beta}, \quad (2)$$

where Q=[MSA]$_o$/[MSA]. For the Diphonix resin, the coefficient β is close to the nominal charge for the eluted ion: for Sr^{2+}, $\beta \approx 1.93 \pm 0.07$, and for Na^+, $\beta \approx 0.99 \pm 0.03$. In the presence of a high salt concentration, k_{ex} decreases, but Eq. (2) still holds approximately, albeit for reduced β. As shown in Figures 4 and 5, this decrease is significant, but the shape of the dependencies for k_{ex} vs. the concentration of the salts added does not depend on the bed ratio. The decrease with the ionic strength of the solution can be described from the empirical formula

$$k_{ex}/k_{ex}^{o} \approx Q^{\beta}/(1 + [\, Q\, [C]/[C]_o]^{\nu}), \quad (3)$$

where [C] is the concentration of the alkali ions (separately). For Sr^{2+}, $[C]_o \approx 19.5$ mM and $\nu \approx 1.25$; for Na^+, $[C]_o \approx 55$ mM and $\nu \approx 1$.

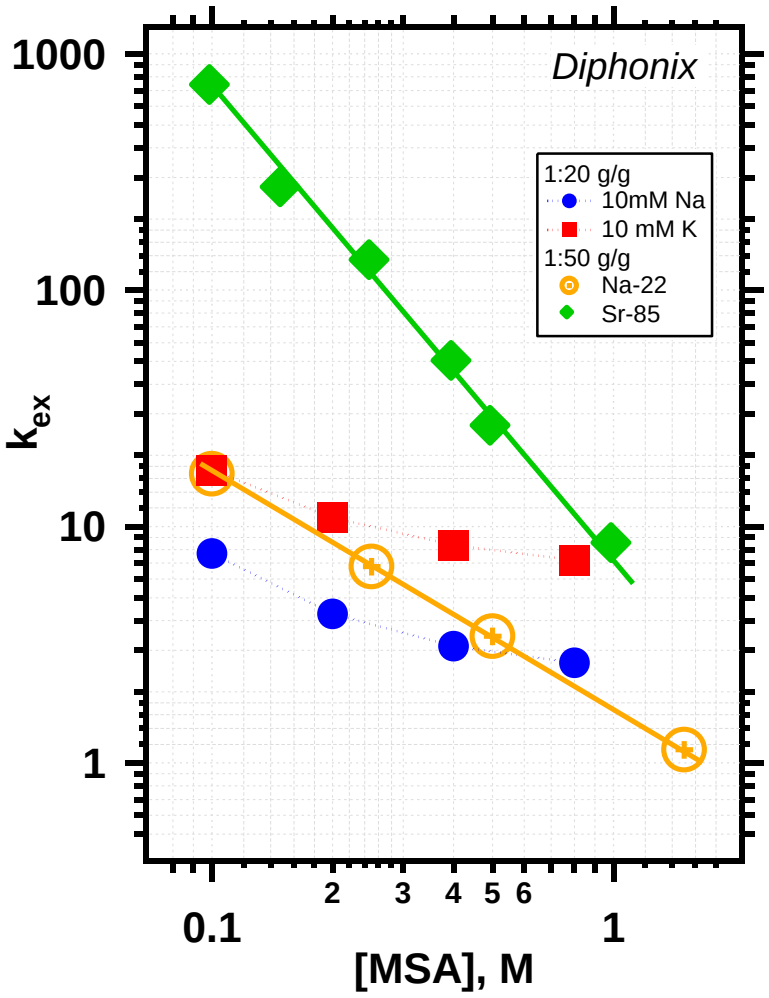

Figure 3. Values of k_{ex} for sodium, potassium, and strontium in an aqueous solution vs. [MSA] for Diphonix resin. Data derived by assuming 0.3 bed ratio and using the data for distribution coefficients. For sodium, data were obtained by using ^{22}Na tracer and inductively coupled plasma optical emission spectroscopy (for 10 mM alkali). The mass ratio for (wet) resin to aqueous solution is indicated in the plot.

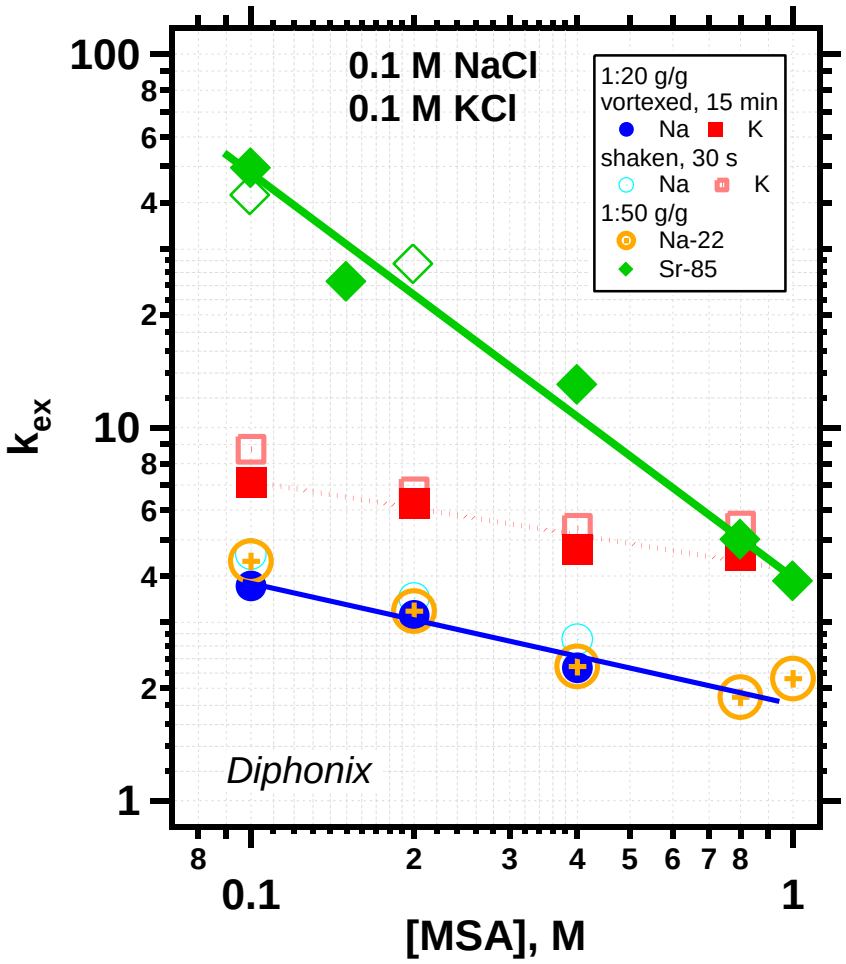

Figure 4. The same as Figure 3, except for an aqueous solution containing 0.1 M NaCl and 0.1 M KCl ("simulated urine matrix"). The distribution coefficients for K and Na were obtained after either agitating the solution for ½ min on an orbital shaker or 15 min of vigorous vortexing.

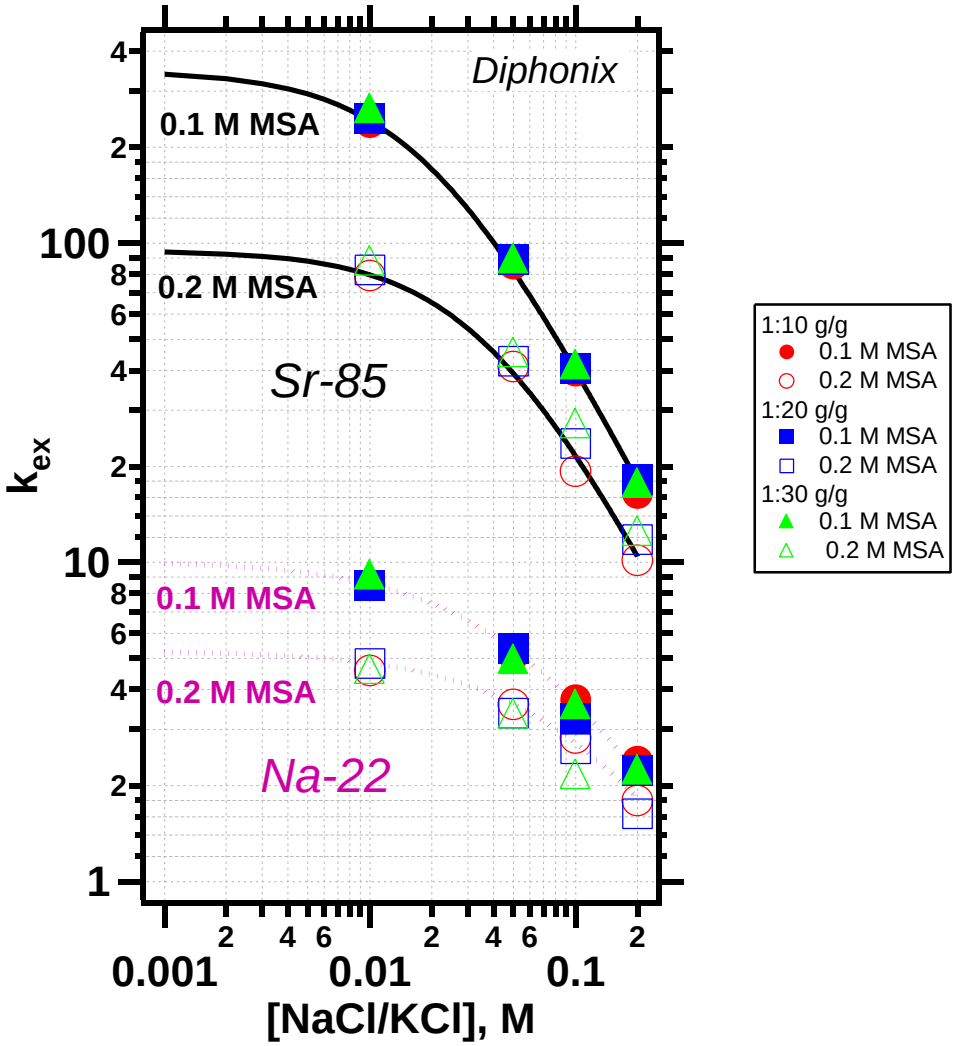

Figure 5. Values of k_{ex} for ^{85}Sr and ^{22}Na extraction with Diphonix resin as a function of the molar concentration of sodium/potassium (1:1) ions in 0.1 M and 0.2 M MSA solutions. The lines are fits to equation given in the text.

A closer look at Figures 4 and 5 reveals that at 0.1 M MSA with the "simulated urine" saline solution containing 0.1 M sodium and potassium chloride, k_{ex} is ≈40 for Sr^{2+} and ≈5 and ≈8 for sodium and potassium, respectively. From these data we calculated that 20-30 volumes can be loaded on the column with a large (> 20) number of theoretical plates without Sr^{2+} breaking through, while Na^+ and K^+ break through the column during this loading. As the acidity dependency for K^+ and Na^+ is less steep than that for Sr^+, increasing the acidity over 0.2 M MSA is not advisable, because the difference in k_{ex} for K^+ and Sr^+ becomes smaller as the acidity increases, and the two ions become more difficult to separate on the column.

Our approach is to load as much sample on the column as possible without Sr^{2+} breaking through the column and then elute K^+ and Na^+ with minimal loss of Sr^{2+}. As shown in Figure 3, for elution with 0.1 M MSA, $k_{ex} < 20$ for K^+ and Na^+, whereas $k_{ex} > 100$ for Sr^{2+}, so the strontium and the alkali ions can be completely separated on a time scale approximately equal to the time required to load the sample on the column. As suggested by the data given in Figure 6, the distribution ratios for ^{85}Sr in a human urine sample are intermediate between the aqueous and the saline solutions. The latter solution represents the worst case scenario (abnormally salty urine).

3. Model Calculations

To simulate the performance of the column, we developed a program to calculate equilibria for each plate of a model column. The coefficients k_{ex} used in these calculations are given in Table 1. Four loading/elution scenarios are considered in Table 2, and the elution profiles are given in Figures 7 to 10. For these simulations, the model column was loaded with 10-30 volumes of 0.1 M NaCl + 0.1 M KCl in 0.1 M MSA, and the alkali ions were eluted using 15-30 volumes of 0.1 or 0.2 M MSA. At the end of the elution, the column was stripped with four volumes of 3 M nitric acid. As seen from these plots and Table 2, 20 volume elution is sufficient to reduce K^+ concentration to < 2 mM in the strip solution, collect > 99% of Sr^{2+}, and achieve a concentration factor between 2.5 and 7.5, depending on the load. The elution can be as high as 30 column volumes, provided that the column has > 20 theoretical plates. For a 10-plate column, 10-20x volume loads can be used.

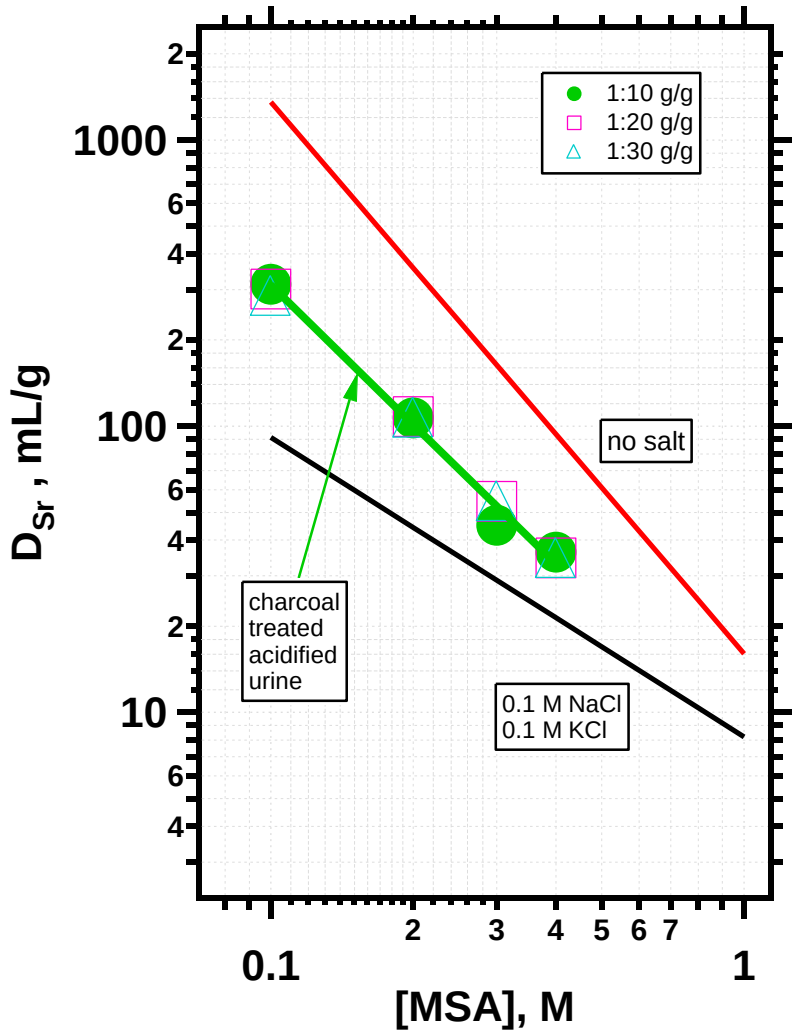

Figure 6. Acid dependence of distribution ratio for strontium-85 on Diphonix resin for urine matrix (symbols), aqueous solution (red line), and 0.1 M NaCl + 0.1 M KCl solution (black line). The distribution coefficients remained constant for 1:10 to 1:30 g/g mass ratio of the wet resin to the liquid sample.

Table 1. Calculated values of k_{ex} for Diphonix and Sr resins (for column operation under equilibrium conditions) using the reported (see http://www.eichrom.com/radiochem/) and measured values for distribution ratios. Bed ratios of 0.30 and 0.25 are assumed for Diphonix and Sr resins, respectively. For the latter resin, the parameters are taken from data supplied by the manufacturer.

Resin	Acid, M	k_{ex}		
		K^+	Na^+	Sr^{2+}
Diphonix + MSA	0.10	17.41	7.71	721.39
	0.15	13.43	5.47	267.00
	0.20	10.95	2.79	182.42
	0.25	9.95	3.98	131.01
	0.40	8.46	3.13	49.25
	0.80	7.21	2.84	13.27
	1.00	-	-	8.36
Diphonix + HNO_3	2.0	-	-	3.81
	3.0			1.44
	4.0			2.87
Diphonix + MSA + 0.1 M NaCl 0.1 M KCl	0.10	7.96	3.98	49.40
	0.15	6.97	3.48	24.53
	0.20	6.47	2.99	24.88
	0.25	5.47	2.74	19.90
	0.40	4.48	2.24	12.99
Sr resin + HNO_3	0.02	0.1	0.10	0.2
	0.05	0.1	0.10	0.7
	0.10	0.9	0.10	1.1
	0.30	1.8	0.20	6.5
	0.50	2.0	0.20	11
	1.00	3.5	0.30	30
	2.00	3.5	0.28	50
	4.00	3.0	0.28	70
	7.00	0.8	0.10	80

Table 2. Results from theoretical modeling of Diphonix column performance under equilibrium conditions. The load solution contains 0.1 M NaCl + 0.1 M KCl in 0.1 M MSA. After elution with 0.1 M or 0.2 M MSA, the column is stripped using 3 M HNO_3.

No.	Fig.	Load, column volumes	No. theor. plates	Eluent, M MSA	Eluted, column volumes	Four column volume strip using 3 M HNO_3		
						Sr, conc. factor	[K], mM	Sr recovery, %
1	7	10	10	0.1	30	2.5	1.3	99.2
2	8	10	10	0.2	20	2.5	0.96	99.4
3	9	20	10	0.2	19	4.95	1.5	99.1
4	10	30	20	0.2	16	7.44	1.7	99.3

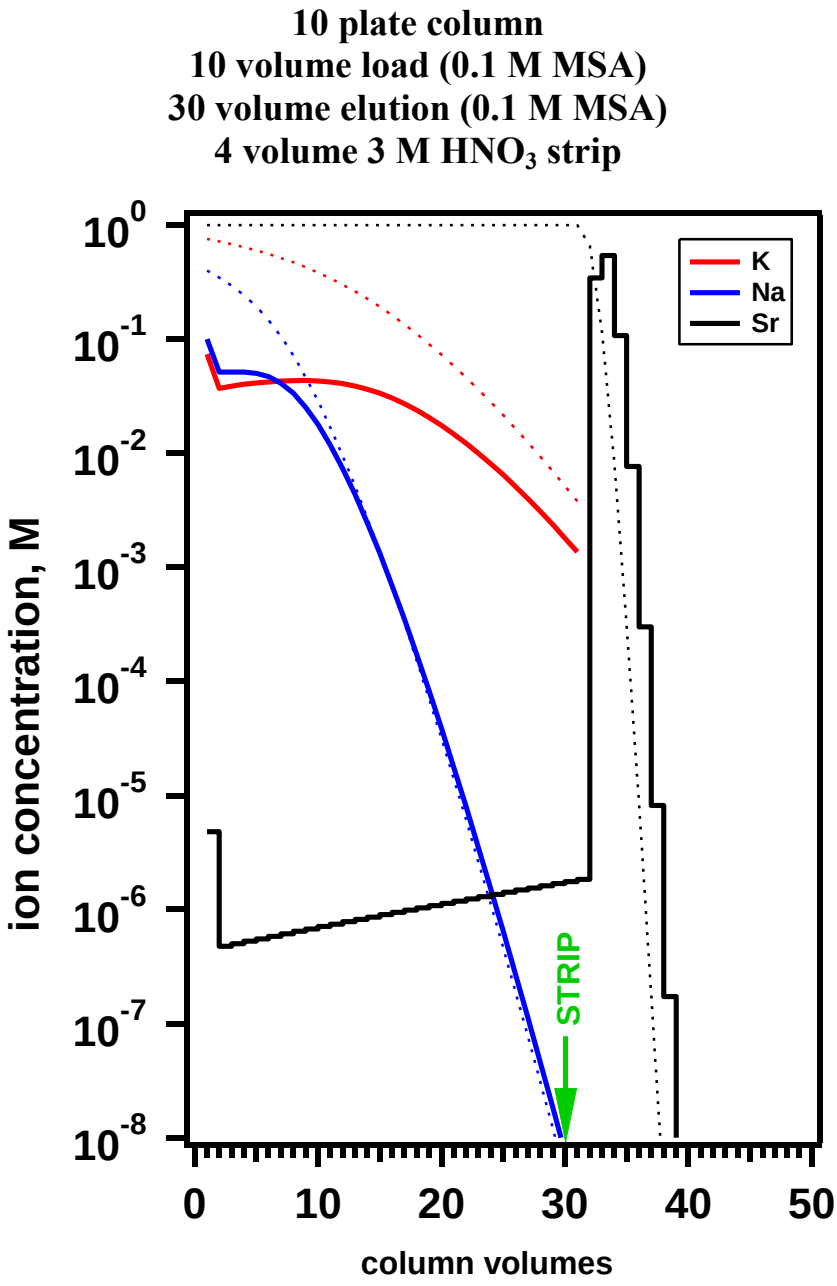

Figure 7. Elution profiles calculated from the data in Table 1 and simulation conditions for test No. 1 in Table 2. Solid lines are concentrations in the eluent, and dashed lines are the average concentrations on the column (both phases).

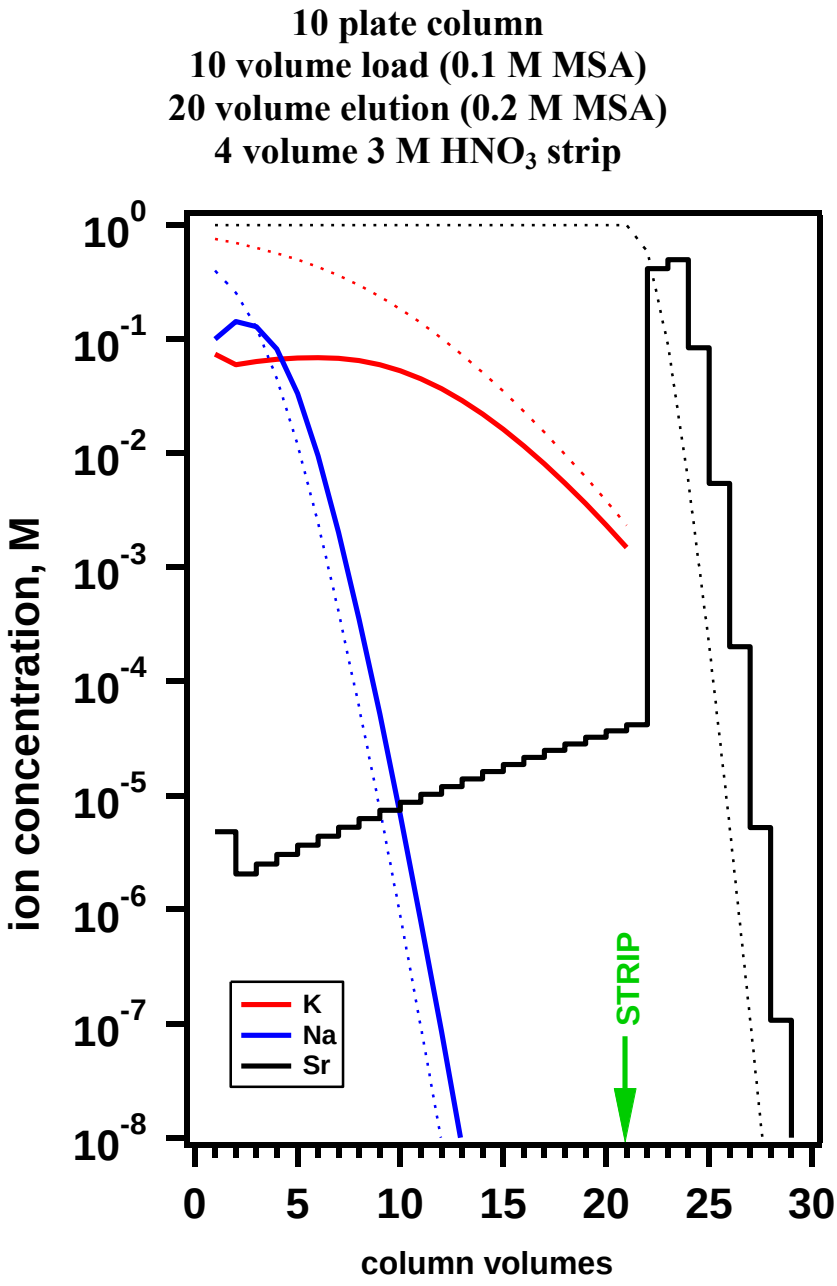

Figure 8. Elution profiles calculated from the data in Table 1 and simulation conditions given for test No. 2 in Table 2.

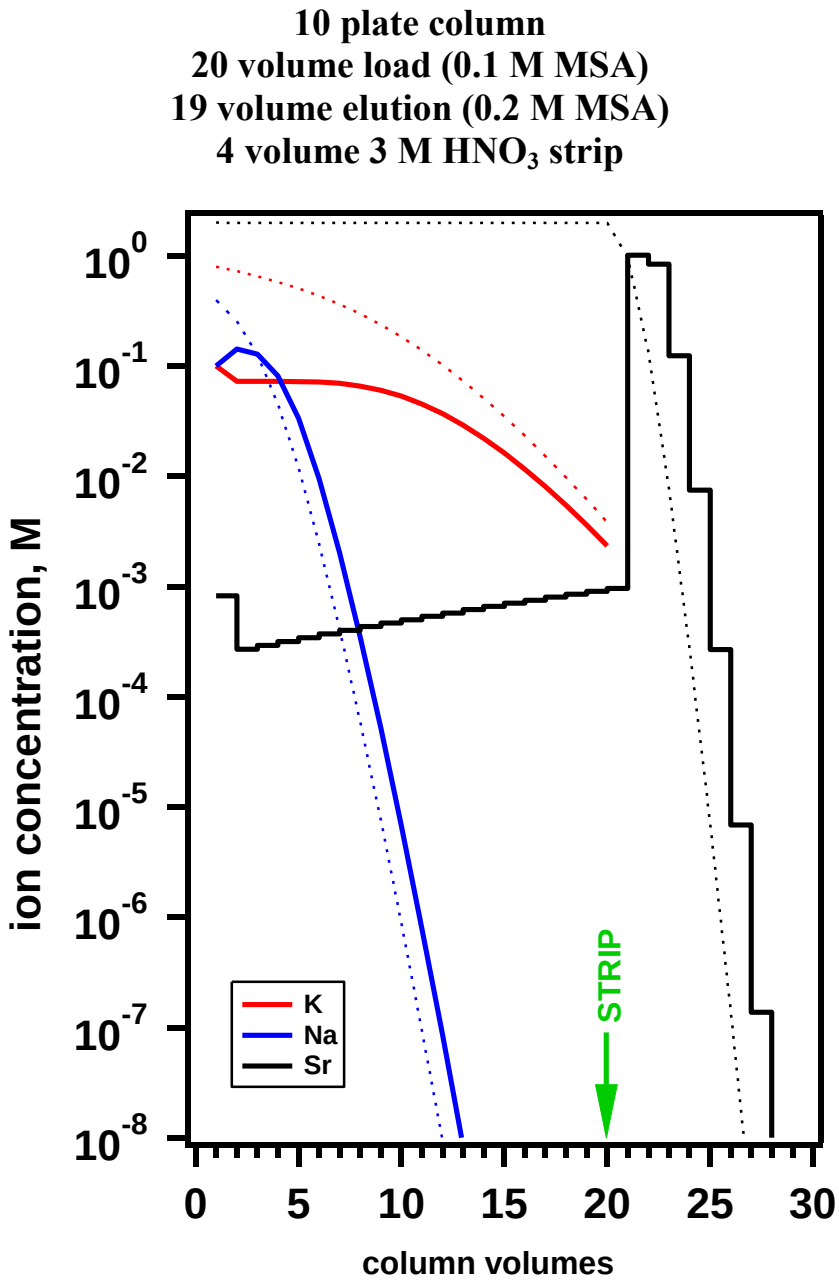

Figure 9. Elution profiles calculated from the data in Table 1 and simulation conditions given for test No. 3 in Table 2.

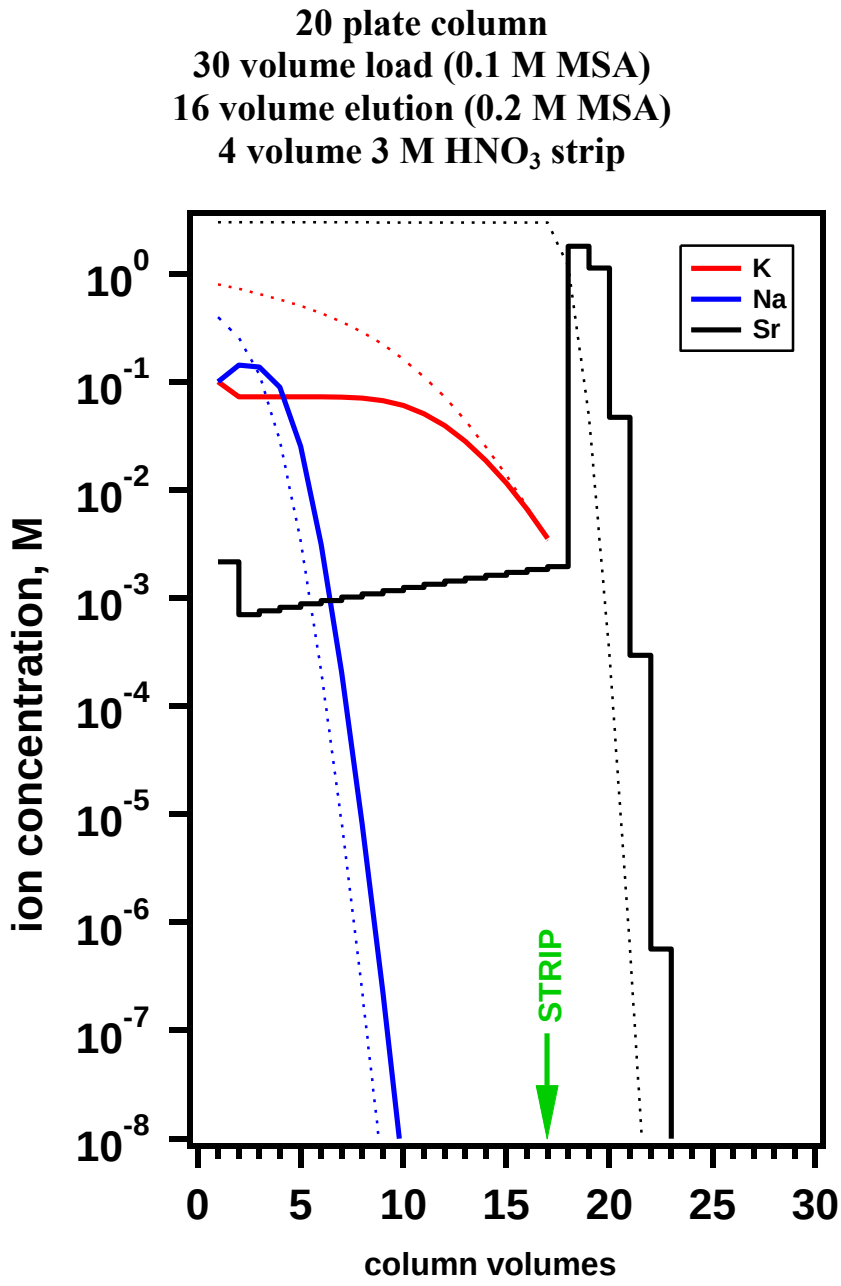

Figure 10. Elution profiles calculated from the data in Table 1 and simulation conditions given for test No. 4 in Table 2.

4. Results and Discussion of Column Tests

The performance of a real column is less predictable since the Diphonic resin column operates far from equilibrium. In the simplest column test (No. 1 in Table 2), 10x volumes of the saline solution in 0.1 M MSA were loaded on a 0.54 cm^2 x 3.7 cm (2 mL) column, then the alkali ions were eluted at 1.5 mL/min, where the eluent was 0.1 and 0.2 M MSA. As shown in Figure 11, the alkalis on the column were eluted down to < 1 mM in 20x volumes (the minimal volumes for elution of each ion are given in Table 3).

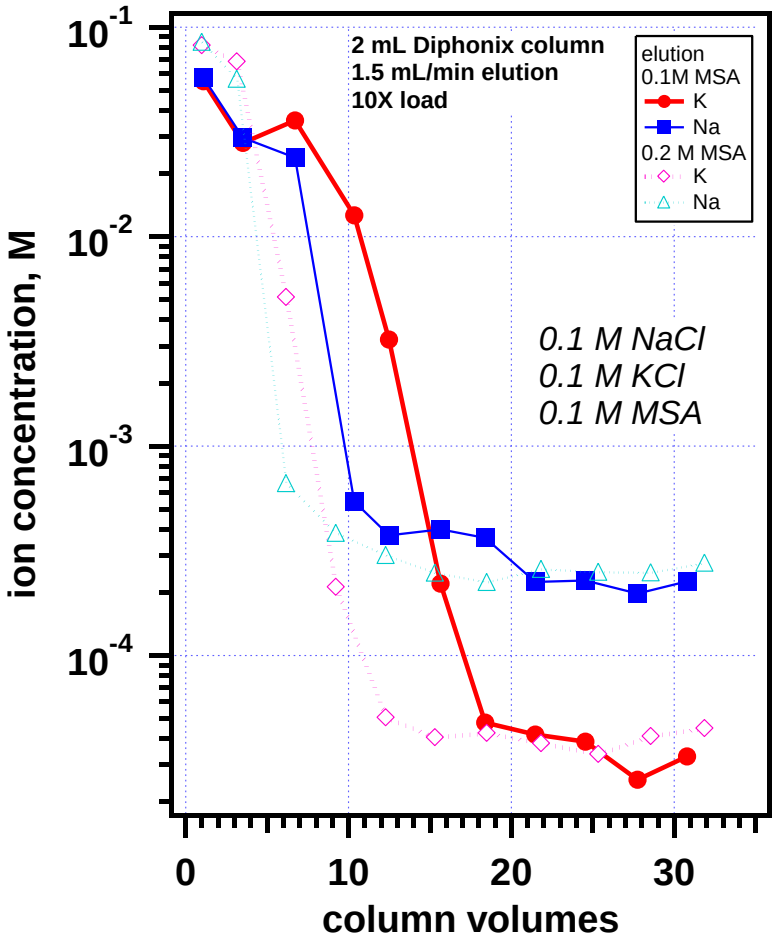

Figure 11. Ion concentrations in the eluent vs. the number of column volumes for test No. 1 (Table 3). Conditions include 10x load of saline solution (0.1 M NaCl + 0.1 M KCl) acidified with 0.1 M MSA (2 mL Diphonix resin column, 0.54 cm^2 x 3.7 cm). The elution is either with 0.1 M MSA (solid lines, filled symbols) or 0.2 M MSA (dashed lines, open symbols).

Table 3. Minimum values of elution for column test No. 1 (Figure 11). Estimates are for the number of column volumes required to elute K^+ and Na^+ ions after 10x column volume load on 2 mL Diphonix resin column (1.5 mL/min elution).

Elution with	0.1 M MSA	0.2 M MSA
K^+	18	12
Na^+	10	7

As shown in Figure 12 and Table 4 for test No. 2, stripping the column after elution of 22x volumes recovered 100% of strontium and reduced the concentration of sodium and potassium to the limits of detectability. In this experiment, only < 40% of the alkali ions was retained on the column during loading, while all strontium ions were retained.

The same relates to test No. 3, with acidified urine (0.1 M MSA) spiked with 6.5 mM strontium (Figure 13 and Table 4). Less than 30% of the alkali ions was retained during 10x load. The alkali ions were eluted down to negligible concentrations in 10x volume elution with 0.1 M MSA, when 98% of strontium was retained at the column. The 15x volume elution resulted in 5% loss of Sr, while the 22 volume elution resulted in 13% loss of strontium; thus, only 87% was recovered in the nitric acid strip following this 22x volume elution. This experiment suggests that an unidentified component of urine decreases the retention coefficients below those in the saline solution, which contradicts the data of Figure 6 for equilibrium conditions. Even though the column operated far from equilibrium, the Sr^{2+} and K^+ were still well separated, and the strontium was largely decontaminated from the alkali cations.

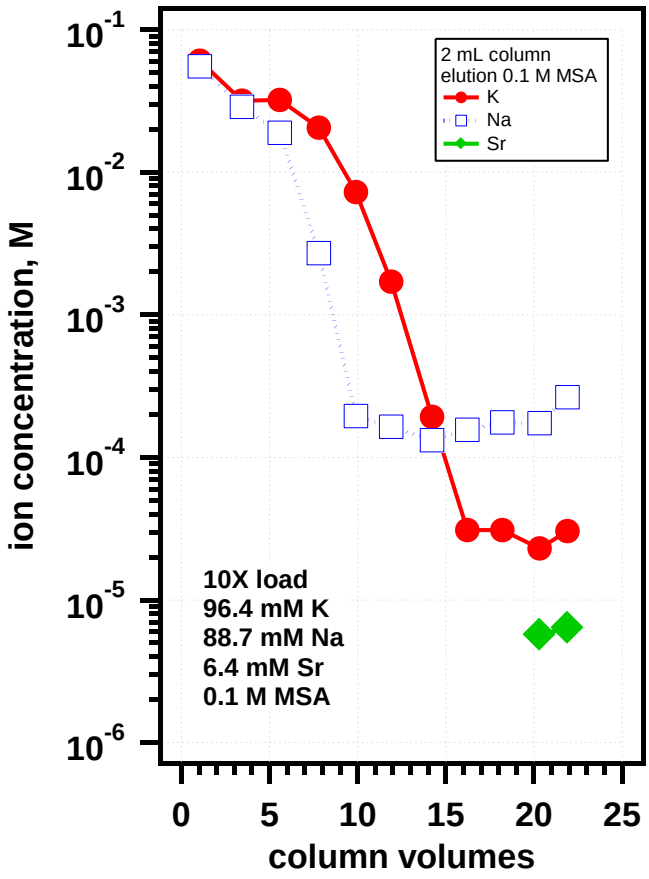

Figure 12. Ion concentrations in the eluent vs. the number of column volumes for test No. 2. Conditions include 10x load of Sr-spiked saline solution acidified with 0.1 M MSA. Ion concentrations in the load are also given in the plot.

Table 4. Trial data for column tests No. 2 and 3 (Figures 12 and 13). Both series are 0.1 M MSA load solutions eluted with 0.1 M MSA. The load was 10 column volumes (2 mL, 0.54 cm^2 x 3.7 cm column) and the elution was to 22-23 column volumes at 1.5 mL/min.

	Column test No. 2 (aqueous matrix) (Figure 12)			**Column test No. 3** (acidified human urine treated with charcoal) (Figure 13)		
	K^+	Na^+	Sr^{2+}	K^+	Na^+	Sr^{2+}
Load, mM	96.4	88.7	6.4	64.2	160	6.4
Load, mmol	1.93	1.77	0.127	1.19	2.98	0.061
Load (effluent), mmol	1.30	1.35	0	0.84	2.28	0
% retained	32.6	23.7	100	29.7	23.5	100
Column, M	0.315	0.21	0.064	0.18	0.35	0.063
Recovered, mmol 3M HNO_3 strip	4.35 $x10^{-5}$	2.75 $x10^{-3}$	0.125	1.45 $x10^{-4}$	2.57 $x10^{-3}$	0.108
Conc. on the column after 22 volume elution	21.5 μM	1.38 mM	63 mM	73 μM	1.29 mM	54 mM
Eluent, mmol by integration	0.56	0.36	-	0.32	0.57	0.017
Sr recovery % after 22 volume elution	100			86.4		
Sr recovery, % after 10 volume elution				98.0		

Figure 14 shows the results of column test No. 4 for a urine sample (10x load, same as for Figure 13) after 15x volume elution using 0.1 M MSA. Approximately 7% of Sr was eluted from the Diphonix resin column, so the recovery of strontium was 93%; however, 99.95% of the Sr^{2+} on the Diphonix resin column was stripped, passing just 3x volumes of 3 M HNO_3. This resulted in 3.2x concentration of Sr^{2+} in the strip eluent. Approximately 82% of Sr^{2+} on the column was eluted with the first column volume of the 3 M nitric acid, generating a very concentrated strontium stream.

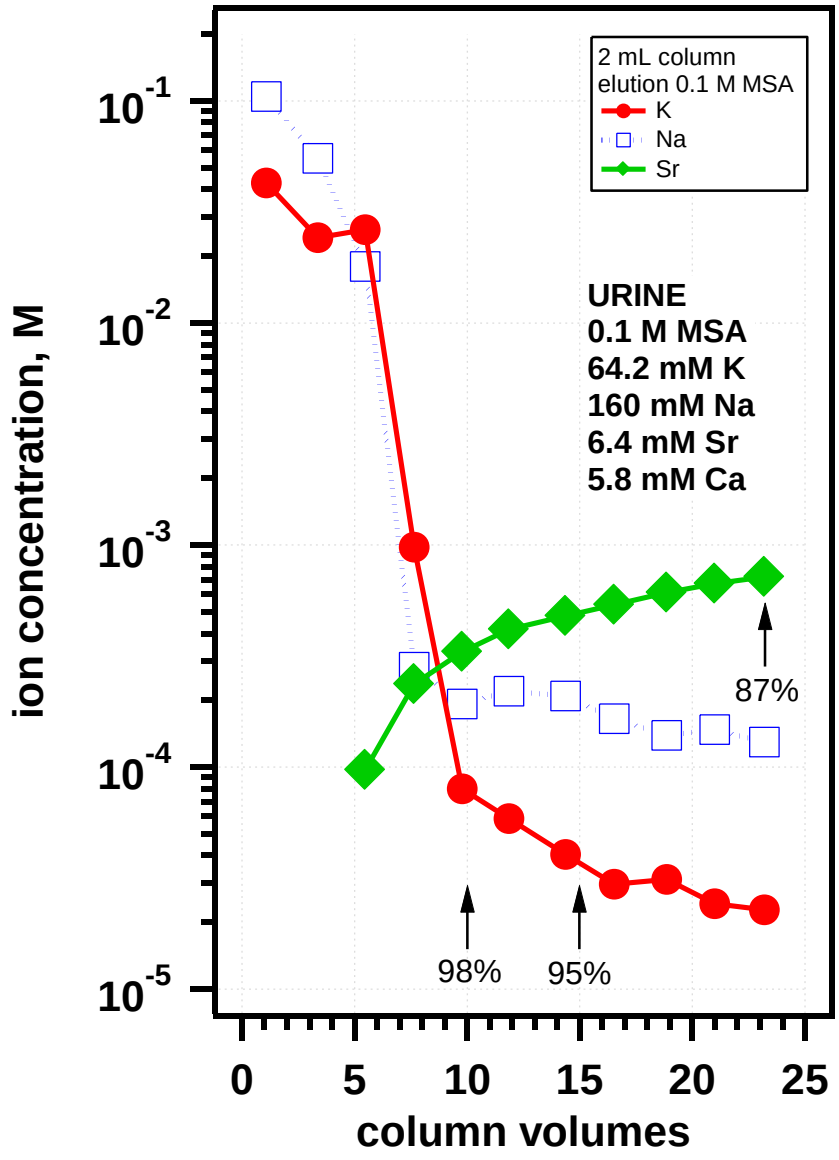

Figure 13. Ion concentrations in the eluent vs. the number of column volumes for test No. 3. Conditions include 10x load of Sr-spiked human urine acidified with 0.1 M MSA. Ion concentrations in the load are also given in the plot.

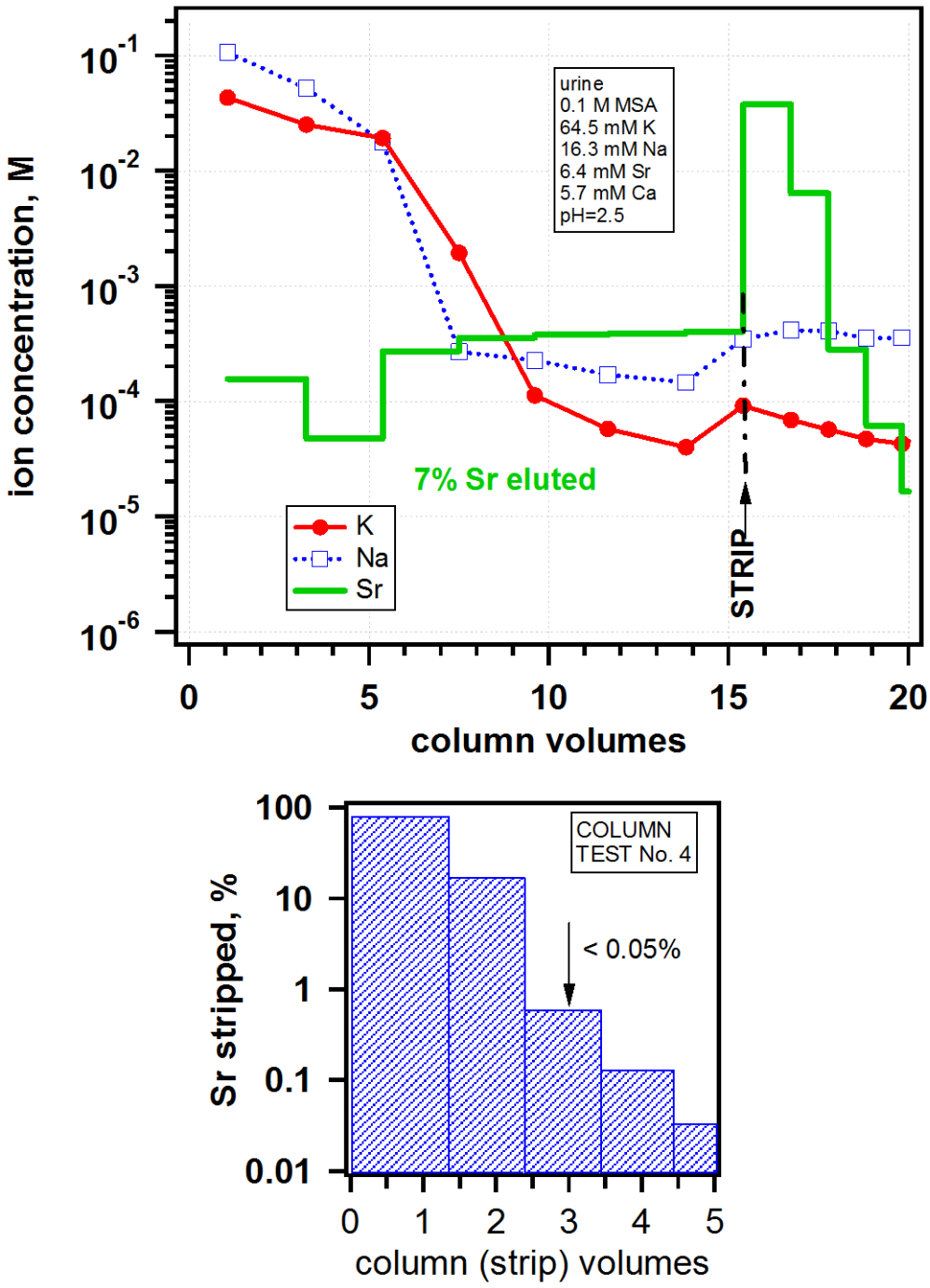

Figure 14. Ion concentrations in the eluent vs. the number of column volumes for test No. 4, as well as Sr percent stripping. Like test No. 3 (Figure 12), Sr was stripped after 15x eluted volumes using 3 M HNO_3. The loss of Sr^{2+} during this 0.1 M MSA elution was 7%; 3x volume stripping of the column using 3 M HNO_3 removed 99.95% of Sr^{2+} on the column. The concentration factor for strontium was 3.2. The concentrations of K^+ and Na^+ in the strip solution were < 1 mM.

Figure 15 presents results from a test on standardized frozen "normal human urine" (Lot IR100706 obtained from Innovative Research, Inc). In this test a 5x volume sample of treated, acidified urine was loaded on 2 mL Diphonix resin columns. In one of the tests, the urine was acidified to pH=1 using 0.22 M MSA (the urine is buffering); in another sample, 0.1 M MSA was added to achieve pH=1.6. There was no loss of Sr during loading and < 0.1% loss of Sr during the elution, in both cases. Stripping the column after 15x eluted volumes of 0.1 M MSA with 3x volumes of 3 M HNO_3 recovered 99.7% of Sr on the column. The concentrations of K^+ and Na^+ in the strip solution were < 0.3 mM (Figure 15).

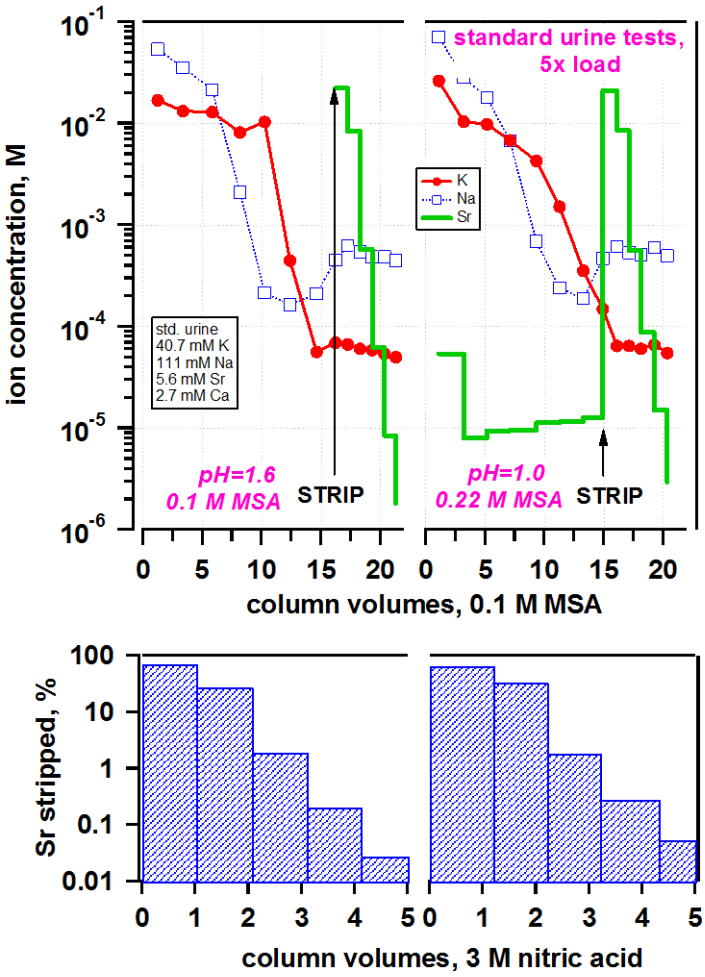

Figure 15. Results from column test on standardized, frozen "normal human urine" obtained from Innovative Research, Inc. (Lot IR100706). The urine sample was acidified with 0.1 M MSA (pH 1.6) or 0.22 M MSA (pH 1) before activated carbon treatment. Conditions: 5x sample volumes were loaded on the 2 mL Diphonix resin column, 15x volumes of 0.1 M MSA were eluted, and the column was stripped using 3 M HNO_3.

The 3 M HNO_3 strip (3x volumes) corresponds to the conditions under which k_{ex} for strontium on the Sr resin is ≈ 60 (Table 1 and Figure 2a). Assuming that the volume of the Sr resin column is 10% of the volume of Diphonix column, we found that loading the strip solution on a 20-plate Sr resin column corresponds to 30x column volumes, resulting in a loss of < 0.1% Sr during the loading. After 5x more volumes of nitric acid to wash away undesirable ions and 2x volume strip using 10 mM nitric acid, Sr is concentrated 20 times in addition to the preconcentration on the Diphonix resin column. This strontium is decontaminated from any residual potassium, calcium, and trivalent ions including yttrium (^{90}Y is the daughter product of ^{90}Sr decay).

5. Proposed Process for Urine Treatment

The above results provide the basis for the suggested process in Figures 16 and 17. At the heart of this process are two columns: a larger column filled with the Diphonix resin and a smaller column filled with Sr resin (which is 10% by volume of the former). To save processing time, while one of the columns is loaded/eluted, the other column is washed/conditioned for the next run (Figure 16). The two columns are connected with a three-way valve. In step 1, the two columns are disconnected, and the Diphonix column is conditioned for loading of Sr^{2+} with 0.1 M MSA while the Sr column is washed with 10 mM nitric acid. In step 2, 10-30x volumes of treated urine (see below) are loaded on the Diphonix resin column while the Sr resin column is still washed by 10 mM nitric acid. In step 3, the alkali ions are eluted using 15-20x volumes of 0.1 M MSA, while the Sr resin column is conditioned for loading by passing 3 M nitric acid. In step 4, Sr^{2+} is stripped from the Diphonix resin column using 3 M nitric acid, and the effluent from the column is loaded directly on the Sr resin column. In step 5, 2x volumes of 10 mM nitric acid are used to strip Sr^{2+} from the Sr resin column, while the Diphonix column is washed with 3 M nitric acid. Following the stripping of Sr^{2+}, the two columns return to the conditions of step 1.

The typical volumes of the two columns are 2 mL and 200 μL, respectively. For a 20 mL load, the first column would concentrate Sr by a factor of 2.5-3.5 and the second column by a factor of 20, resulting in an overall concentration factor >50. We expect that the whole process takes one hour. Higher concentration factors are possible for the Diphonix resin columns with 30-40 theoretical plates. We have not yet optimized the design of the columns and the operation conditions.

This analytic procedure requires pretreatment of the urine sample (see Figure 17). The pretreatment we settled upon involves the acidification of urine with 0.1 M MSA (introduced in neat form) followed by stirring of the acidified solution with 1 wt% activated carbon. After this treatment (< 5 min), the sample is centrifuged at 3500 rpm for

15 min, and the supernate is filtered through a 0.15-0.45 μm polypropylene filter to remove microparticles of activated carbon.

This treatment achieves several objectives. At pH < 2, Sr^{2+} is released from the carriers that bind it in urine. Unlike nitric acid, MSA does not react with bile pigments, forming strong light-absorbing products that interfere with beta-counting. Activated carbon removes pigments and high-molecular weight compounds that foul the ion-exchange column. Our tests indicate that activated carbon does not retain Sr^{2+} during the treatment, provided that pH < 2. Since the pH of urine naturally varies from 4.4 to 8 and urine has mildly buffering properties, addition of 0.1 M MSA may not result in pH <2 for some samples. In this case, the pH should be adjusted with MSA until the pH < 2.

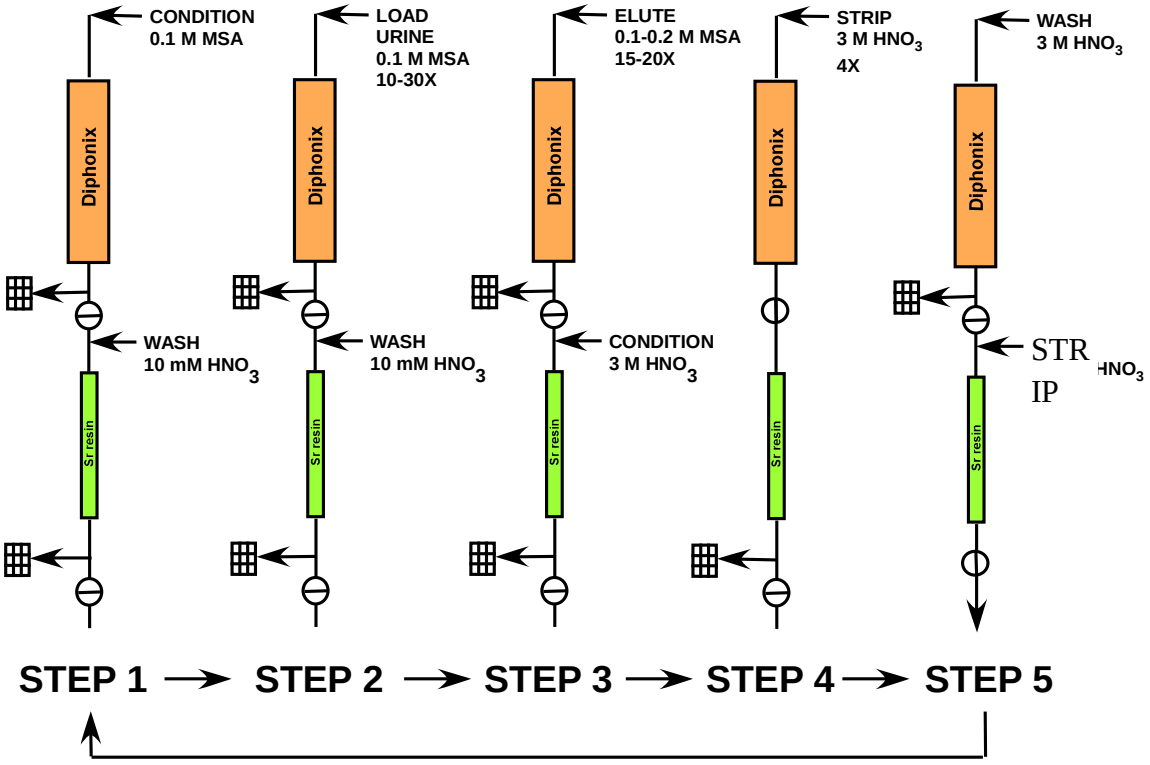

Figure 16. Scheme for ^{90}Sr extraction, decontamination, and concentration using the tandem Diphonix–Sr resin column, as described in the text. The overall treatment scheme is considered in Figure 17.

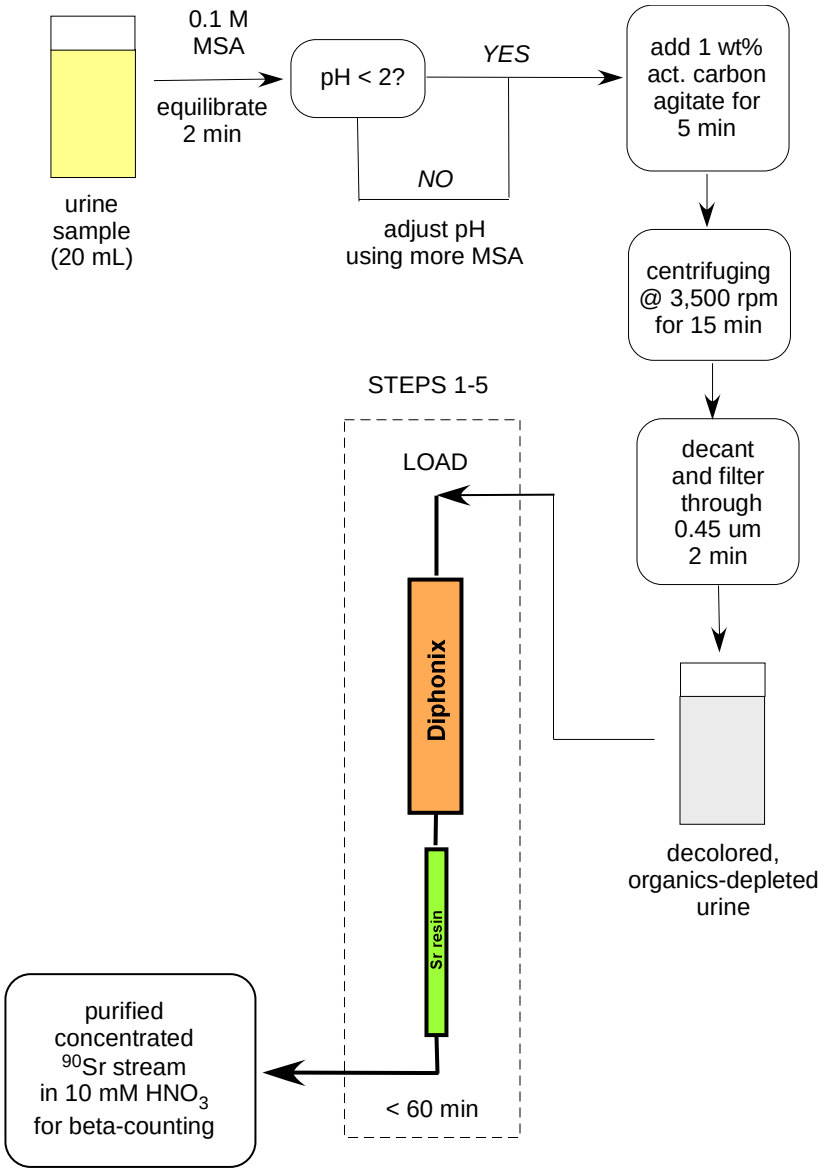

Figure 17. Proposed scheme for rapid extraction, decontamination, and concentration of ^{90}Sr from human urine samples. Steps 1 to 5 are shown in Figure 16.

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